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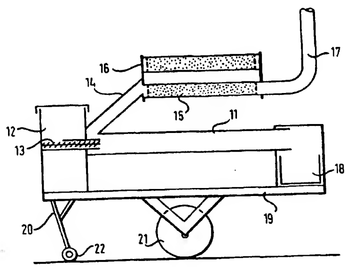
(54) Title: IMPROVEMENTS IN THE PRODUCTION OF BIOGENETIC SILICA FROM ORGANIC PLANT MATTER



(57) Abstract

In a method for the production of biogenetic silica from organic plant matter, the plant matter is fed continuously by a screw conveyor (13) from a hopper (12) to a furnace in the form of an inclined tube (11) which is maintained at a temperature of about 450° and rotated at a speed of about 1 rpm. The material travels down the inclined tube and is retained in the furnace for no more than about 30 minutes. Biogenetic silica is collected from the lower end of the tube (11) in a collection can (18) and is allowed to cool. The inclined tube (11) may be mounted on a wheeled platform (19) so that the furnace is portable.

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Title: IMPROVEMENTS IN THE PRODUCTION OF BIOGENETIC SILICA FROM ORGANIC PLANT MATTER

This invention relates to improvements in the production of biogenetic silica from organic plant matter and is particularly concerned with the production of ash from rice hulls or husks, rice stalks, equisetum (commonly known as horsetails), palm leaves and in particular those of the palmyra palm as well as sunflowers and bamboo.

The disposal of rice husks is a substantial problem for rice growers since the husks are not suitable for use as fertilisers and up until now have had to be disposed of either by burying or burning. Space for burying is frequently not available in heavily populated areas and burning creates undesirable atmospheric pollution. However, as is known, rice husks contain a high amount of biogenetic silica and it is possible to produce pozzolanic material from rice husk ash which has great potential economic advantages for developing countries that have large quantities of rice husks readily available and virtually free of cost.

Attempts have previously been made to utilise rice husks mixed with Portland cement to make blocks. It has generally been possible to meet strength requirements but the cup-like form of the husks requires an excessive amount of cement and thus such blocks are not economic. Accordingly, it has been proposed to reduce the rice husks to ash form for use in the production of pozzolanic material.

In the conversion of rice husks to ash, the combustion process removes the organic matter and leaves a silica rich residue. When rice husks are first heated, weight loss occurs up to 100^oC by evaporation of absorbed water. At about 350^oC, the volatiles ignite causing further weight loss and the husks begin to burn. From 400^oC to 500^oC, the residual carbon oxidises and the majority of weight loss occurs in this period. The silica in the ash is still in amorphous form. Above 600^oC, some conversion to quartz occurs.

In previous work carried out on rice husk ash, the ash was produced by burning large quantities of rice husks in a heap in a furnace, often for long periods. Under these conditions, the optimum burning regime had been found to be a temperature of 450^oC for 3 hours. The energy requirement under these conditions makes production uneconomic for most, if not all, commercial applications. Similar problems are encountered with the combustion of other organic plant matter of the kind referred to above.

The present invention therefore seeks to improve the production of rice husk ash to enable it to be produced economically on a commercial basis and the primary aim of the invention is to provide an improved method of producing biogenetic silica from organic plant matter.

According to the invention there is provided a method for the production of biogenetic silica from organic plant matter in which the plant matter is fed continuously to an inclined rotating furnace maintained at a temperature of between 400 and 500^oC, the material

is retained in the furnace for no more than about 30 minutes and biogenetic silica is continuously collected from the furnace and allowed to cool.

Preferably, the rotating furnace is maintained at a temperature of about 450^{o}C and the material is desirably retained in the furnace for between 15 and 30 minutes.

The organic plant matter is desirably fed into the upper end of the inclined rotary furnace via a hopper. The biogenetic silica, which normally takes the form of ash, may be collected from the lower end of the furnace via a chute.

The organic plant matter may be ground to a powder before being fed to the furnace.

According to a preferred embodiment of the invention, the organic plant matter takes the form of rice husks or rice hulls and the biogenetic silica takes the form of ash.

The invention will now be explained and described in detail with reference to the drawings, in which:-

Fig. 1 is a schematic diagram of a furnace for conducting tests for the production of rice husk ash;

Fig. 2 is a graph showing the grading curve for ground rice husks;

Figs. 3-7 are graphs showing the temperature-time relationships for tests on rice husks using the furnace shown in Fig. 1; and

Fig. 8 is a diagrammatic representation of a furnace suitable for the continuous production of rice husk ash by the method according to the invention.

Tests have been carried out on rice husks using the tube furnace 1 shown in Fig. 1 of the drawings which had a heated length of 700mm and a tube of 75mm inner diameter. A ceramic lining tube 2 of 60mm inner diameter was then set up within the furnace and was supported by a friction drive arrangement (not shown) to allow rotation. In order to retain material in the central section (approx. 300mm) of the tube during combustion, two loosely fitting discs 3, 5 were inserted on the ends of respective tubes 4, 6. Air was passed through the tube 4 as indicated by the arrow and a thermocouple 7 was inserted in the other tube 6.

Rice husks were obtained from Tarragona and tests were carried out on ground and unground husk. The grading of the ground husks is shown in Figure 2 of the drawings. The bulk densities of the ground and unground husks were 513 and 107 kg/m^3 respectively. Two temperatures and two different combustion times were chosen and in all five tests were conducted as follows:-

		Target temp.	Time
Test 1	Ground husk	450°C	30 min
Test 2	Ground husk	750°C	60 min
Test 3	Ground husk	450°C	60 min
Test 4	Ground husk	750°C	30 min
Test 5	Unground husk	450°C	60 min

The upper target temperature was limited to 750°C in order to ensure that no overshoot above the acceptable limit could occur.

The tube was first charged with the husk and the tube allowed to rotate for a short period in order to distribute the material uniformly. A speed of rotation of 1 rpm was adopted. The furnace was then set to the target temperature. Both the temperature in the furnace windings and the inner tube were monitored and minor adjustments made as necessary during the course of the tests.

In order to ensure a free tumbling effect, the tube was filled to a depth of no more than about one-third of its diameter. For the ground husk roughly 90g was introduced. In the case of the unground husk it was necessary to place a greater than ideal bulk in order to ensure a reasonable return; 42g was introduced in this case.

After maintaining the furnace at the set temperature for the required period it was allowed to cool. For the tests conducted at 450^{o}C it was possible to withdraw the inner tube immediately and allow it to cool protected against excessive cooling rates, which might have damaged it, by an insulating blanket or a foil shield. At the higher temperature it was not possible to remove the inner tube and it had to be left overnight to cool.

The ash was then raked from the tube and material which was retained in the central section throughout the combustion was separated from that which had been transported further along the tube.

Although the temperature rise of the furnace was quite rapid, it has to be accepted that the ash was held at a high temperature for an additional period whilst cooling

was allowed. This was not a serious problem at the lower temperature, but must clearly be allowed for in interpreting the findings from the high temperature tests.

Figures 3-7 show the temperature-time relationships for the tests and the main events occurring during the tests are indicated on these graphs together with the 30 or 60 minute hold period. The furnace temperature and the temperature in the lining tube are shown.

The furnace temperature was controlled by a thermocouple in the furnace windings and clearly the temperature in the inner tube would be lower than this until equilibrium had been reached, with the possible exception of the temperature being raised temporarily due to the combustion itself. The furnace was initially set for a temperature 50°C above the target and minor adjustments made in the later stages as necessary.

It was found that the temperature recorded by the thermocouple in the lining tube was lowered by about 70°C by the passage of air through the tube and this must be allowed for when interpreting the results.

For handling reasons it was necessary to introduce the husk into the furnace cold and then raise the furnace temperature. As can be seen from the graphs, this occurred fairly rapidly, reaching 500°C within 15 minutes. Early in the heating, water vapour was driven off and smoke emission commenced when the temperature in the lining tube reached 180°C. At this stage the air

supply was turned on. During the combustion phase there was a rapid rise in temperature in the lining tube, briefly overtaking the furnace temperature in tests 3, 4 and 5. Combustion was completed within 10-20 minutes, which, in some cases, was before the target temperature was reached.

With the ground husk, some fine, partly burned particles were carried out with the air flow, but the unground husk was retained within the tube.

After burning for the required time, the ash was removed from the tube taking care to separate material which had been transported out of the central section during combustion and using only the material which had been retained in the central section for subsequent analysis.

The analysis of the ash samples was in three parts:

(a) Determination of carbon, hydrogen and nitrogen content,

(b) EDAX analysis to determine the amounts of silicon and metallic elements present,

(c) X-ray diffraction analysis to establish the forms of silica present.

The results of these analyses were as follows, with contents expressed as percentages by weight:

Element	1C	2C	3C	4C	5C
C	2.10	1.48	2.14	1.35	2.10
H	0.19	-	0.93	0.10	0.19
N	-	-	-	-	-
Si	87.48	86.01	86.47	84.84	93.46
K	4.98	4.92	6.27	5.97	1.26
Fe	0.87	0.35	0.63	0.86	0.01
Cu	4.49	4.33	2.94	3.72	2.37
Zn	2.18	2.35	2.59	1.99	1.60
Ca	-	2.04	1.11	2.62	1.30

The X-rd report on silicon dioxide crystallinity found the following:

Sample 1C Almost completely amorphous; small amount (fraction of a percent) of unidentified material, possible a feldspar.

Sample 2C Approximately 1% cristabolite; traces of unidentified material (as 1C) and possibly quartz.

Sample 3C As 1C.

Sample 4C As 2C.

Sample 5C Completely amorphous.

It is thought that the traces of unidentified material relates to contamination from a previous use of the grinder. This would be consistent with its possible identification as a feldspar. The same is true of the higher potassium and, to a lesser extent, copper contents of the ground husk ash samples. The grinder had previously been used for clay and soil samples and there is the possibility that contamination occurred.

All samples of ash produced satisfy the requirement of a high amorphous silica content. Hence the principle that a usable ash can be produced at low temperatures and in a short time is established on a small scale. The retention time in the furnace after combustion had no advantageous effect on the end product and could probably be reduced further.

The key factor is to ensure that husks or husk particles are kept separated from each other as far as possible during the combustion, achieved here by the use of a rotating tube furnace with a small charge of material. If too much material is introduced there is a risk that the temperature will rise excessively due to the exotherm during combustion. This is clearly indicated in Test 5, in which a greater volume of material was used and there was a very sharp rise in temperature during combustion. If the temperature reached becomes too high then the crystalline content of the ash will increase.

Pre-grinding of the husk before burning does not appear to offer any advantage in the light of the short combustion time involved. In terms of final ash composition, the unground husk gave the best result.

There are, however, other advantages to be gained from grinding beforehand arising from the increase in the bulk density, namely reduction in transportation costs and permitting a greater furnace throughput. The ground particle size used in these tests was probably smaller than the optimum as some particles were small enough to be transported through the furnace in the airflow despite the fact that this was kept at the lowest possible level.

The tests indicated that rice husk ash could be produced in a relatively short time if the furnace was maintained at 450°C under the right conditions and thus made the ash production on a commercial basis an economic possibility. If the rice husks can be moved through a furnace on a continuous basis and agitated while they are heated, heating times can be reduced to 15 to 20 minutes.

Accordingly, the furnace shown diagrammatically in Fig. 8 of the drawings is employed. As shown in Fig. 8, the furnace comprises an inclined tube 11 which is mounted on support stands (not shown) for rotation about its longitudinal axis by suitable drive means (likewise not shown). The furnace is provided substantially throughout its length with heating elements (also not shown). The furnace is further provided, at or adjacent to its upper end, with a hopper 12 through which rice husks, which may be ground, are fed to the furnace by means of a screw feeder 13. A smoke outlet 14 leads from the tube furnace 11 to a filter 15 which is mounted in a rotating holder 16 and the filtered air is led from the filter 15 to an air extraction system (not shown) via a pipe 17. The end of the furnace tube 11 remote from the feed hopper 12 is provided with an ash

collection can 18 at which the ash is collected. The hopper 12, furnace tube 11 and ash collection can 18 are mounted on a platform 19 which can be tilted by means of an adjusting jack 20 to ensure that the hopper 12 is located at a level which is higher than that of the can 18 so that material fed to the furnace tube 11 will tend to pass down the tube from the hopper 12 to the can 18. The platform 19 is further desirably provided with wheels 21 whereby the apparatus is portable and the adjusting jack 20 is desirably also provided with similar but smaller wheels 22.

The furnace is desirably rotated at a speed of about 1 rpm and is set at a temperature of 450°C. The retention time of the rice husks in the furnace is determined by the rate of rotation of the tube 11 and the angle of declination of said tube. However, under ideal conditions it should be possible to collect ash from the can 18 some fifteen minutes after the rice husks enter the furnace from the hopper 12. Thus a continuous production of rice husk ash on a commercially economic basis is assured.

The ash has a high amorphous silica content and can be used for the production of dense concrete and aircrete blocks. Because of the good combustion conditions, the ash also has a low carbon content (approximately 2% by weight) which is a highly desirable feature because a higher level of carbon reduces ash reactivity.

Although the invention has been described above with reference to the production of ash from rice husks it should be noted that the invention is not restricted to the production of rice husk ash but is applicable generally to the production of biogenetic silica from organic plant matter.

CLAIMS

1. A method for the production of biogenetic silica from organic plant matter in which the plant matter is fed continuously to an inclined rotary furnace maintained at a temperature of between 400 and 500°C, the material is retained in the furnace for no more than about 30 minutes and biogenetic silica is continuously collected from the furnace and allowed to cool.

2. A method according to claim 1, wherein the rotary furnace is maintained at a temperature of about 450°C.

3. A method according to claim 1 or claim 2, wherein the material is retained in the furnace for between 15 and 30 minutes.

4. A method according to any preceding claim, wherein the organic plant material is fed into the upper end of the inclined rotary furnace via a hopper.

5. A method according to any preceding claim, wherein the biogenetic silica is collected from the lower end of the inclined rotary furnace via a chute.

6. A method according to any preceding claim, wherein the organic plant matter is ground to a powder before being fed to the furnace.

7. A method according to any preceding claim, wherein the organic plant matter takes the form of rice husks or rice hulls and the biogenetic silica takes the form of ash.

8. Apparatus for performing the method claimed in any one of the preceding claims, said apparatus including a furnace which comprises an inclined tube (11) mounted on support stands for rotation about its longitudinal axis, means for rotating the inclined tube about its said longitudinal axis and a plurality of heating elements located in the furnace substantially throughout the length of the tube; a hopper (12) for receiving organic plant matter; means for feeding the organic plant matter from the hopper to the furnace at one end of the tube and means for collecting combusted material from the other end of the tube.

9. Apparatus according to claim 8, wherein the means for feeding organic plant matter from the hopper (12) to the tube (11) comprise a screw conveyor (13).

10. Apparatus according to claim 8 or claim 9, wherein the means for collecting combusted material comprise a collection can (18).

11. Apparatus according to any one of claims 8 to 10, wherein the furnace is provided with a smoke outlet (14) which leads to a filter (15) mounted in a rotating holder (16) and leading to an air extraction system.

12. Apparatus according to any one of claims 8 to 11, wherein the furnace is mounted on a platform (19) having means for adjusting the angle of inclination of the platform and hence of the tube.

13. Apparatus according to claim 12, wherein the adjusting means comprise an adjusting jack (20).

14. Apparatus according to claim 12 or claim 13, wherein the platform is provided with wheels (21) whereby the apparatus is portable.

15. Apparatus according to any one of claims 8 to 14, wherein the means for rotating the tube are arranged to rotate the tube at about 1 rpm.

FIG. 1

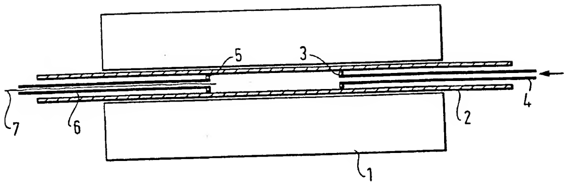

5
3
4
2
7
6
1

FIG. 2

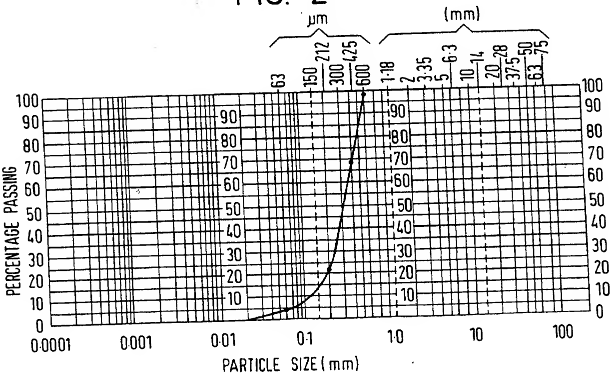

µm
(mm)
63
150
212
300
425
600
1·18
2
3·35
5
6·3
10
14
20
28
37·5
50
63
75
PERCENTAGE PASSING
PARTICLE SIZE (mm)
0·0001
0·001
0·01
0·1
1·0
10
100

FIG. 3

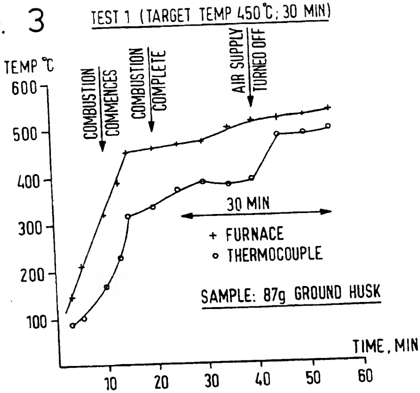

TEST 1 (TARGET TEMP 450°C; 30 MIN)
TEMP °C
600
500
400
300
200
100
COMBUSTION COMMENCES
COMBUSTION COMPLETE
AIR SUPPLY TURNED OFF
30 MIN
+ FURNACE
o THERMOCOUPLE
SAMPLE: 87g GROUND HUSK
TIME, MIN
10
20
30
40
50
60

FIG. 4

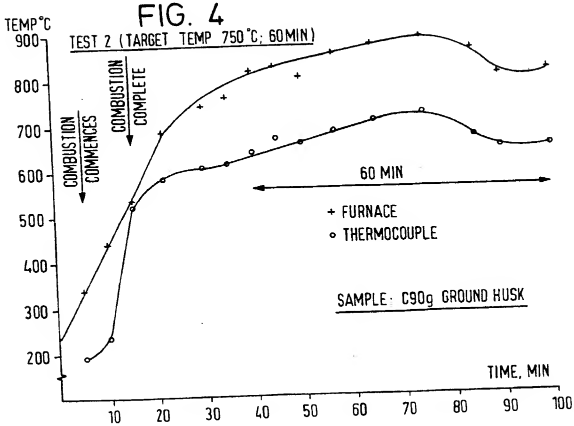

TEMP °C
900
800
700
600
500
400
300
200
TEST 2 (TARGET TEMP 750°C; 60MIN)
COMBUSTION COMMENCES
COMBUSTION COMPLETE
60 MIN
+ FURNACE
o THERMOCOUPLE
SAMPLE: C90g GROUND HUSK
TIME, MIN
10
20
30
40
50
60
70
80
90
100

FIG. 5

TEST 3 (TARGET TEMP 450 °C; 60 MIN)

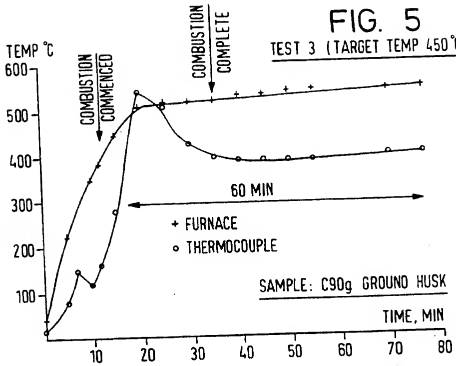


FIG. 6

TEST 4 (TARGET TEMP 750 °C; 30 MIN)

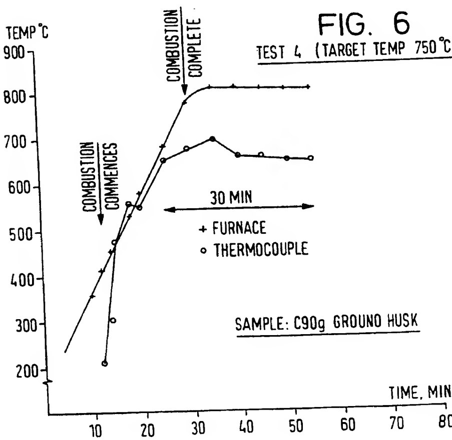
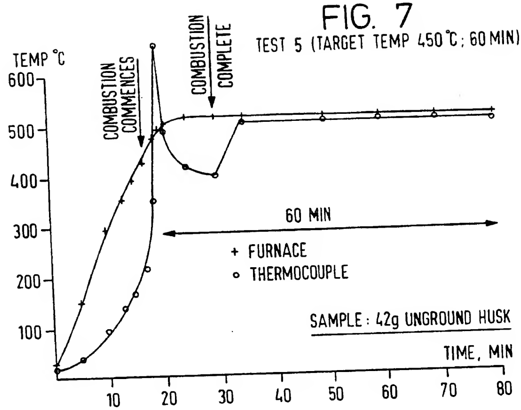


FIG. 7


TEST 5 (TARGET TEMP 450°C ; 60 MIN)
TEMP °C
600
500
400
300
200
100
COMBUSTION COMMENCES
COMBUSTION COMPLETE
60 MIN
+ FURNACE
o THERMOCOUPLE
SAMPLE : 42g UNGROUND HUSK
TIME, MIN
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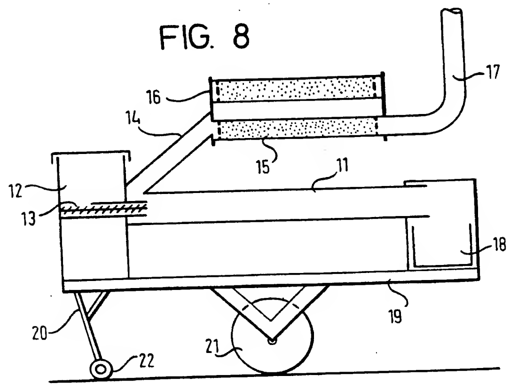
20
30
40
50
60
70
80

FIG. 8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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 94/02106

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C04B18/10 F23G5/40 F23G7/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C04B F23G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CHEMICAL ABSTRACTS, vol. 113, no. 4, 23 July 1990, Columbus, Ohio, US; abstract no. 28326m, A.F.GALAL ET AL. 'Rice husk ash in portland blended cement' see abstract & SILIC. IND., vol.55, no.1-2, 1990 page 55-8	1,2,7-11
Y	US,A,4 037 543 (J.F.ANGELO) 26 July 1977 see column 7, line 4 - line 24; claims	1,2,7-11
A	US,A,4 049 464 (A.TUTSEK ET AL.) 20 September 1977 see column 3, line 35 - column 4, line 5 see column 5, line 46 - line 65 see column 7, line 49 - line 56	1,7

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"&" document member of the same patent family

1

Date of the actual completion of the international search	Date of mailing of the international search report
23 December 1994	10. 01. 95
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Theodoridou, E

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Intern. ...al Application No
PCT/GB 94/02106

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	V.M.MALHOTRA 'Fly Ash, Silica Fume,Slag, and Natural Pozzolans in Concrete; Proceedings 4th International Conference, Istanbul, Turkey, May 1992' May 1992 , ACI S.SUGITA et al. : 'Evaluation of Pozzolanic Activity of Rice Husk Ash' Volume I see page 495 - page 512 ---	1-7
A	DE,A,25 59 527 (L.&C. STEINMÜLLER GMBH) 31 March 1977 see claims ---	1-7
A	US,A,4 730 564 (H.I.ABBOUD) 15 March 1988 see claims; figure 1 ---	8-15
A	US,A,2 127 328 (F.J.EGAN) 16 August 1938 see the whole document ---	8-14
A	GB,A,969 538 (MOUNT ROYAL RICE MILLS LTD) 9 September 1964 -----	1,2,7

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

Intern al Application No
PCT/GB 94/02106

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
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US-A-4049464	20-09-77	DE-A-	2416291	23-10-75
		FR-A,B	2266672	31-10-75
		GB-A-	1508825	26-04-78
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		JP-B-	56031286	20-07-81
		SE-B-	394886	18-07-77
		SE-A-	7503649	06-10-75
DE-A-2559527	31-03-77	NONE		
US-A-4730564	15-03-88	NONE		
US-A-2127328		NONE		
GB-A-969538		NONE		